

Editorial

Special Issue: Phenolic Profiling and Antioxidant Capacity in Agrifood Products

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Phenolic compounds are secondary plant metabolites known to be one of the most important sources of natural antioxidants in the human diet. These compounds play important roles in long-term health and reducing the risks of chronic and degenerative diseases. Apart from the biological capacities shown by phenolics in in-vivo and in-vitro studies, they present protective effects against the deterioration of foods and beverages because of their intrinsic nature as antioxidants. For all these reasons, the search for new sources of natural antioxidants, nutraceuticals and functional foods have been the subject of many studies in recent years. However, such compounds are potentially vulnerable to different factors of plant processing (such as light, temperature, pH, oxygen, etc.) for obtaining different food and beverage products. Consequently, substantial modifications to their structure and concentration could occur, leading to changes in their potential biological activities. Recent endeavors have placed particular importance on finding plant-processing methods and techniques for stabilizing plant-based products that do not alter their phenolic content and therefore their antioxidant and other biological activities. This Special Issue aims to bring together the most recent work, on the one hand, on the development of new functional food and nutraceutical products with high phenolic content and antioxidant potential, and on the other hand, on the impact that conventional and advanced food processing technologies [e.g., pulsed electric fields (PEF), pulsed-light (PL), ultraviolet (UV)-light; high pressure processing or high hydrostatic pressure (HPP/HHP); ultrasound; extrusion technology, etc.] have on the phenolic and bioactivity characteristics of industrial foods.

This Special Issue of *Processes*, entitled “Phenolic Profiling and Antioxidant Capacity in Agrifood Products” (https://www.mdpi.com/journal/processes/special_issues/phenolic_antioxidant (accessed on 27 June 2022)), includes seven recent experimental studies of interest and two reviews by leading researchers in the topic. The studies include the analysis of different extraction and identification techniques involved in the recovery of bioactive substances from agrifood products, and the study of the in-vivo and in-vitro bioactivity of compounds present in different plant matrices.

The different published papers successfully cover the subject of this Special Issue and we believe that they are highly relevant for researchers in the field, the agrifood sector and the general public. Below is a summary of the objectives, results, discussions and conclusions obtained for each of the different works published in this SI.

The work of González-Romero et al. [1] involved the study of bioactive compounds [total carotenoids (TCC) and flavonoids (TFC) contents, and chlorophyll pigments (a and b)] from different extracts (methanolic, lipophilic and hydrophilic fractions) of Spanish moringa leaves (MO). In addition, the following methods were used to determine their antioxidant capacity: total antioxidant capacity (TAC); ferric ion reducing antioxidant power (FRAP); 2,2'-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid (ABTS)); 2,2-diphenyl-1-picrylhydrazyl inhibition (DPPH) and oxygen radical absorbance capacity (ORAC) assays.



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The total polyphenol content (TPC) was studied following the Folin–Ciocalteu method. The importance of the study arises from the interest in promoting the healthy composition of this plant, widely consumed fresh or cooked in soups or porridges in different continental regions such as Africa, the Middle East, south-east Asia, and the Pacific, Caribbean and South America. It is considered to be a good alternative/complement to the packaged salad leaves commonly consumed nowadays. The results were compared with 28 leaves in pre-packaged salads. The MO leaves showed the highest TAC values among the extracts analyzed and the richest contents in TPC, TCC, TFC and chlorophylls, indicating that the addition of this fourth-range ingredient to fresh-cut salad foods would result in up to a six-fold increase in the antioxidant content of this commercial product that currently provides over 50% of the antioxidant capacity of all the vegetables consumed in Spain.

Král et al. [2] studied the addition to biscuits of different concentrations (1–10%) of a selected herb (mint), spices (cinnamon and cloves) or fruit (grape) extracts and their influence on the content of polyphenols (TPC by Folin–Ciocalteu), antioxidant capacity (FRAP and DPPH), and their sensory properties and attractiveness to consumers. The results showed that the use of appropriate spices and herbs in the elaboration of the biscuits increased their polyphenol content and thus their antioxidant capacity, with the highest values found in biscuits elaborated with the highest amount of cloves. However, at this level of addition, the products were not well-accepted by consumers, presenting a reduction in texture (hardness and fracturability) dependent on the amount of spices/herbs added, and a different color compared with the control sample. Therefore, the authors concluded that the addition of cloves at a concentration of 3.0% appears to be the best option to obtain biscuits with a combination of increased antioxidant capacity (AC) and overall consumer acceptability. The authors stated that adding a reasonable quantity of natural substances containing natural antioxidants improves the overall quality of biscuits.

Ordóñez-Díaz et al. [3] studied the bioactive composition (phenolic compounds, anthocyanins and organic acids) by HPLC-DAD-HRMS, and antioxidant activity by ABTS, DPPH, and ORAC assays, of oranges from two cultivars, ‘Salustiana’ and ‘Sanguinelli’, widely used in the orange juice industry. Both cultivars were grafted on four different citrus rootstocks, namely Forner-Alcaide no.5, ‘Cleopatra mandarin’, Citrus volkameriana and Carrizo citrange, to evaluate their influence on the chemical composition. The potential interest to the citrus industry and consumers of these citrus rootstocks lies in the fact that they are widely used to guarantee high quality and production levels of oranges. Furthermore, the influence on chemical composition and bioactivity of the intra-year variation (2016/2017 and 2017/2018) and harvest period (from December to April) were evaluated.

The results showed that the ‘Sanguinelli’ cultivar presented the highest levels of flavonoids, mainly hesperidin and narirutin, and the anthocyanins cyanidin-3-O-(6''-malonyl) glucoside, followed by cyanidin-3-O-rutinoside and cyanidin-3-O-glucoside. The main organic acids were citric acid, followed by malic, oxalic, and ascorbic acids. Furthermore, the fruits from the Forner-Alcaide no.5 rootstock showed the highest amounts of compounds such as phenolics and organic acids and the highest antioxidant capacity values. This suggests that it is a good option for the citric sector because of its positive effect on the health-promoting properties of fruits obtained from both cultivars studied. Regarding the effect of the harvesting period, fruits harvested at the beginning of the season (January) presented the maximum contents in carotenoids and organic acids (mainly citric acid), while the phenolic compounds and antioxidant activity reached the highest levels between February and April.

Concerning the harvesting year, the antioxidant activity was higher in the second year in both cultivars. The authors highlighted that the information provided may be useful for the juice industry as a starting point for the development of high-quality orange juices with significant levels of bioactive compounds.

Noore et al. (2022) [4] investigated the influence of different extraction strategies, including conventional extraction and novel technologies such as ultrasound-assisted extraction (UAE), microwave-assisted extraction (MAE), pulsed electric field (PEF), high

pressure processing (HPP), and enzyme-assisted extraction (EAE), on the recovery of phenolic compounds (liquid chromatography-mass spectrometry LC-MS, and Folin–Ciocalteu method) and associated antioxidant properties (DPPH and FRAP) from buckwheat hull (*Fagopyrum esculentum*). Forty-one phenolic compounds belonging to eleven different groups including anthocyanins, flavanols, flavanones, flavones, flavonols, phenolic acids, isoflavones, lignans and quinones were identified in the extracts. Lignan and quinone groups were found in high amounts, while the anthocyanin and flavone contents were low. However, anthocyanins and flavones were only identified in samples extracted using novel strategies, whereas conventional and control samples were limited to lignan and quinone groups of phenolic compounds.

The results suggested that the extraction parameters and strategies employed played a major role in enhancing the amount of phenolic compounds extracted from buckwheat hull. The samples extracted using HPP had the highest extraction yield and antioxidant properties. However, the highest content of TPC was recorded in EAE samples. The authors suggested further studies to enhance the extraction level of phenolics and other parameters, such as sensorial and color properties, to confirm the suitability of buckwheat hull extracts for food formulation applications.

Merinas-Amo et al. [5] investigated the possible health benefits of two lyophilized ale beers (blond and stout), as well as two of their most abundant bioactive compounds, namely tyrosol and iso-alpha humulone. The authors conducted a set of in-vivo assays to evaluate the toxicity, antitoxicity, mutagenicity, antimutagenicity, lifespan and healthspan using *Drosophila melanogaster*. Moreover, they used in-vitro assays using the cancer cell line HL-60 to determine the epigenetic modulation of DNA, the clastogenic activity and the tumor cell inhibition growth. The results showed that all the products studied were safe, neither toxic nor genotoxic. Moreover, all the assays suggested that the key activities of blond and stout ale beers are linked to the modulation of degenerative processes via protection against the oxidative genotoxine H_2O_2 , the extension of lifespan in *D. melanogaster*, the induction of clastogenic proapoptotic DNA damage in HL-60 cells and DNA fragmentation and the modulation of the methylation status. Furthermore, the beer bioactives, tyrosol and iso-alpha humulone, showed different activities depending on the assay and on the concentration used. All these results highlighted that the biological benefits attributed to moderate beer consumption cannot be assigned to any specific bioactive compound due to the high variability and complexity of the phenolic profile as well as the multifactor brewing process including the raw materials, yeast strains, wort composition and fermentation conditions. To sum up, this study shed light on the potential benefits for health of moderate daily beer consumption.

López-Belchí et al. [6] studied the promising value-added use as a functional ingredient of waste of Tintorera grapes, a type of *Vitis vinifera* L. used as a wine coloring in the wine-making industry. The investigation focused on obtaining anthocyanin-rich formulations for the food industry, employing different spray-drying (inlet temperatures of 90 °C and 120 °C and outlet temperatures of 50 and 80 °C) and extract (storage time for one month, and temperatures of 20 and 40 °C) conditions. The results showed that this type of grapes contains large amounts of anthocyanins, mainly malvidin 3-O-hexoside, in addition to presenting antioxidant capacity and the potential inhibition of enzymes involved in pathological conditions of energy metabolism (lipase and glucosidase) and inflammatory and cognitive decline problems (acetylcholinesterase). Furthermore, the encapsulation of anthocyanin content with maltodextrin (10% w:v and 90 °C) offered protection against incident light and inlet temperature allowing the stabilization of the extracts for 4 weeks. The authors highlighted the promising alternative industrial management of this wine-making waste as a sustainable and innovative recovery strategy of bioactive compounds from Tintorera grapes and their use as a rich bioactive extract in new beverages and foods.

Ordóñez-Díaz et al. [7] evaluated the phenolic content (the profile by HPLC-DAD-ESI/MS and TPC by Folin–Ciocalteu method) and the antioxidant capacity of strawberries (*Fragaria × ananassa* Duch., cv. Primoris) cultivated under abiotic stresses, namely a re-

duction in the nitrogen percentage and salinity concentration of the irrigation water, to avoid the contamination of aquifers and using the adverse conditions existing in Almeria (south-eastern Spain) for the irrigation water. The fruit profile showed nineteen phenolics, mainly ellagitannins, anthocyanins and flavan-3-ols. The results showed that the harvesting date was the factor with the greatest influence on the parameters studied, the intermediate sampling date (March) standing out with regard to bioactive content. Furthermore, the reduced nitrogen supply increased the antioxidant capacity of the fruits, while the salinity improved the levels of flavan-3-ols, anthocyanins, and the total phenolics. The authors concluded that these plant growth conditions did not have a negative impact on the phytochemical quality of the fruits and the Primoris variety showed good stability to the studied stress conditions.

Lampakis et al., 2021 [8] reviewed the emerging technologies for the recovery of functional compounds from by-products derived from pomegranate (*Punica granatum*) peels as an environmental prospect with economical value. Specifically, the authors evaluated the bioactive compounds extracted with methods such as simple stirring, pressure-applied extraction, enzymatic extraction, ultrasound-assisted extraction, pulsed electric fields, high hydrostatic pressure, ohmic heating, microwave assistant extraction and the use of new, inexpensive, non-toxic (“green”) solvents such as deep eutectic solvents. The authors observed that the studies highlighted the importance of choosing the ideal solvent and the correct design equipment and extraction method to improve extraction efficiency. They concluded that compared to conventional techniques (simple stirring, decoction, and maceration) that provide low disruption ability of the cell walls and, consequently, low diffusion of the solvents used for the extraction, more recent extraction methods achieve larger-scale rupturing of the cell wall, leading to increased extraction efficiency. Moreover, the combination with a vacuum enhances the protection against oxidation and thermal degradation of sensitive bioactive components. Finally, the authors highlighted the need to adapt most of the developed novel green extraction methods tested so far on a laboratory-scale reactor to an industrial scale for commercial applications.

Rodríguez-Solana et al. [9] composed a review of different publications about the potential uses of carob (*Ceratonia siliqua* L.) pulp, a by-product of the carob seed processing industry, as a functional ingredient in different food preparations. This publication highlighted the chemical properties of this part of the plant, characteristic of the Mediterranean regions, as a good source of macro- and micronutrients such as carbohydrates, vitamins and minerals, and secondary metabolites with functional properties. In addition, its different bioactive properties were also extolled, as well as its potential use as a healthier alternative to cocoa because of its similar sensorial, chemical and biological properties, low-fat content, and the absence of the stimulating alkaloids theobromine and caffeine. Moreover, this publication highlighted the role of the exploitation of this fruit as a whole in rural regions with a Mediterranean climate and scarce agronomic resources. This exploitation could result in the preservation and recovery of the plant *Ceratonia siliqua* L., enhancing economic activity in underdeveloped regions.

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